

X-ray Diffractometer

# XRD-6100



## Ease of use and abundant functions herald a new era of analysis

The Windows 7-supported application software ushers this compact, multi-functional, general purpose X-ray Diffractometer into the networking era of analysis.

X-Ray Diffractometer

Lab  XRD-6100



In addition to its basic ease of use and abundant functions, the XRD-6100 boasts an integrated design featuring a vertical goniometer and data processing software supporting the Windows 7 user interface. The XRD-6100 offers solutions encompassing wide-ranging analysis requirements, from routine qualitative and quantitative analysis to state change analysis, including stress analysis, residual austenite quantitation, crystallite size/lattice strain, crystallinity calculation, materials analysis via overlaid X-ray diffraction patterns, enhanced material evaluation and sample heating analysis. Of course, crystalline structural analysis, including precise lattice constant determination, is also supported.

# A general-purpose X-ray diffractometer to address your various analytical needs XRD-6100

## Features

### **High-precision built-in vertical goniometer**

Enables measurements of a variety of samples including hard-to-secure samples like powders and thin films as well as highly soluble samples.

### **Windows 7 employed as software platform**

The main unit control and data processing software supports the widely used Windows 7 user interface. For this reason, data can export to marketed software. Network support and multi-user accessibility are easily achieved.

### **Multi-functional auto-search/match software (qualitative analysis) equipped as standard**

The XRD-6100 is equipped with auto-search / match software as standard to aid qualitative analysis - the important analysis task of X-ray diffraction. The detailed search parameter settings, second search function, and the comparison display of candidate substances on a raw data profile make analysis easy to understand - even for beginners. What's more, a greater success ratio in results can be achieved with the system. In addition, an easy quantitative calculation function and a function that incorporates element data from the X-ray fluorescence spectrometer are included in the system.

### **Routine performance maintenance is easy**

Adjustments to the optical system are performed by automatic setting function. And since the XRD-6100 has a function to automatically save the system's adjustment parameters, system status can be monitored and recorded. Consequently, routine performance maintenance can be easily controlled, which is vital for quality control in analytical instruments.

### **Safe, compact body**

The main body has been massively slimmed and as the rear is a sheer flat surface with no superfluous protrusions, the device can be placed up against walls, which means it does not take up room when installed on site or in the lab. The unit has also been specifically designed to promote ease of handling in sample loading and positioning and, together with the door lock mechanism engaged during X-raying, provides a safe operating environment.

# Applications of X-ray Diffractometry

## Ferrous metals

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### Steel

Qualitative analysis of steel sheet, measurement of residual austenite and residual stress, analysis of friction and wear test samples, measurement of iron oxide films and nitride layers, evaluation of plating and texture.

### Cast iron

Qualitative analysis of precipitates and additives in cast iron.

### Surface-treated steel

Evaluation of characteristics of surface-treated areas, quality control, residual stress measurement.

## Non-ferrous metals

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### Copper and zinc

Qualitative analysis of alloys, orientation measurements of foil samples, evaluation of texture, qualitative analysis of plated areas.

### Aluminum

Qualitative analysis of aluminum and aluminum alloys, oxides and nitrides, evaluation of texture in rolled material.

### Other metals

Qualitative analysis of metal alloys, oxides and nitrides, evaluation of characteristics of surface-treated areas, residual stress measurement.

## Machinery, automobiles, shipbuilding

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### Machinery

Qualitative analysis of tool steels, surface analysis of machined parts, analysis of austenite layers, qualitative analysis of surface plating, residual stress measurement.

### Automobiles and shipbuilding

Qualitative analysis of structural parts, quantitative analysis of austenite, residual stress measurement.  
Qualitative analysis of exhaust gas catalysts.

## Chemicals and catalysts

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### Chemicals

Qualitative analysis of organic and inorganic chemicals, analysis of impurities.

### Catalysts

Qualitative analysis and degree of crystallinity measurements, measurement of crystallite size to check final product.

## Ceramics

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### Porcelain and ceramics

Qualitative analysis of raw materials, final product evaluation, analysis of crystal structures during heating (crystal system, crystallite size, lattice constant).

### Cement and glass

Qualitative and quantitative analysis of clinker and cement (free lime, etc.), qualitative analysis of raw materials. Qualitative analysis and orientation measurements of thin film layers formed at the glass surface.





## Pharmaceuticals and medical treatment

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### Pharmaceuticals

Qualitative analysis of raw materials, identification of impurities. Crystal polymorphism analysis and degree of crystallinity measurements, quality control during pharmaceutical manufacture using crystallite size measurement. Final product quality check and crystal polymorphism analysis related to patents.

### Dental materials

Qualitative analysis of dental materials such as apatite.

## Resources and energy

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### Coal, oil, natural gas

Plant-scale qualitative analysis, evaluation of carbon materials, evaluation of catalysts during petroleum refining.

### Rocks and minerals

Qualitative analysis of raw materials and identification of impurities. Qualitative/quantitative analysis of asbestos minerals (compatible with PRTR method).

## Electrical and electronic materials

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### Electrical components

Qualitative analysis of corrosion and adhering matter on electrical contacts. Stress measurements in structural parts, qualitative analysis of plated parts.

### Electronic components

Qualitative analysis and orientation measurements of thin-film electronic materials. Measurement of substrate crystal orientation for magnetic heads and electronic elements.

### Superconductors

Crystal structure analysis of superconducting materials using the Rietveld method.

## Construction and civil engineering

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Qualitative/quantitative analysis of asbestos in construction materials. Qualitative analysis of construction materials such as tiles and bricks.

## Environment and industrial wastes

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### Environment

Qualitative/quantitative analysis of asbestos in the work environment. Qualitative analysis of dust.

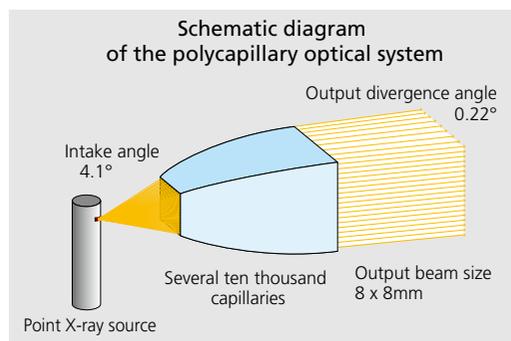
### Industrial waste

Qualitative analysis of residual matter in plating liquids, combustion ash, coal ash, and furnace slag.

# Polycapillary optical system

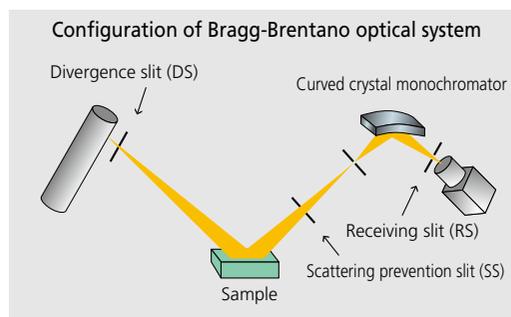
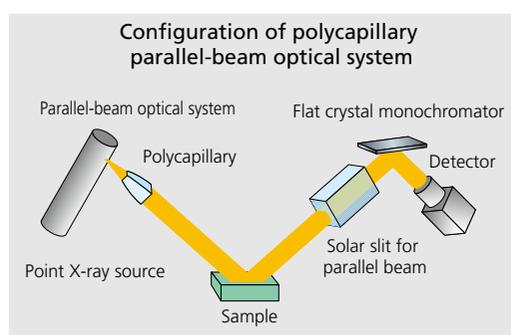
## Principle of the polycapillary optical system

The fine glass capillaries in the order of several microns are arranged in a solid as guides to multiple X-rays. The X-rays pass along each capillary while repeating total internal reflection and exit from the opposite end of the polycapillary system. The capillaries are curved so that repeated total internal reflection is allowed, and the X-rays from the point X-ray source exit the unit as a parallel beam with a large solid angle.



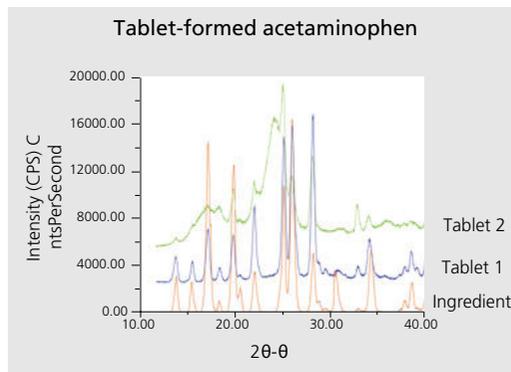
## Features of the polycapillary optical system

Compared with the conventional focused beam system and the normal parallel-beam system, the polycapillary optical system more efficiently exploits the beam from the X-ray tube, resulting in higher diffraction X-ray intensity. A displacement of the sample in a Bragg-Brentano optical system can move it outside the focus, causing a significant displacement in diffraction angle and a dramatic drop-off in diffraction X-ray intensity. Conversely, a displacement of a few millimeters in a parallel-beam system has no effect on the diffraction angle and a small decrease on the diffraction X-ray intensity. Consequently, incorrect loading of the upper and lower sample faces or an uneven sample surface causes no angular displacement and accurate measurement is possible. The parallel-beam system also allows analysis of curved surfaces, something not possible with conventional optical systems.



## Sample measurement using the polycapillary optical system

This example shows measurements of the raw drug acetaminophen and its tablets during the process of manufacturing. Tablets can be directly analyzed to evaluate the degree of crystallinity and crystal polymorphism. The XRD-6100 is able to perform accurate, highly sensitive measurements on irregular surfaces or curved surfaces like this.

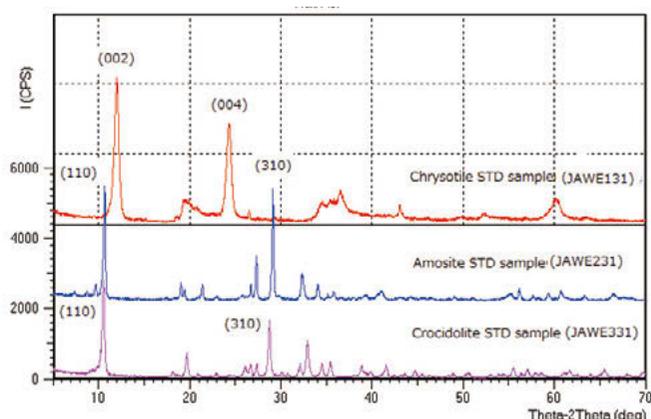


# Qualitative and quantitative analysis of asbestos and free silicic acids

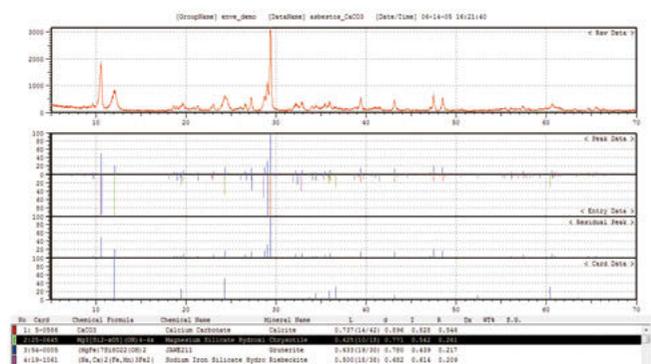
The concentration of asbestos in construction materials is measured using phase contrast dispersion staining microscopes and X-ray diffractometers.

After pulverizing the sample acquired from the site in a pulverizer, a phase contrast dispersion staining microscope and X-ray diffractometer are used respectively to qualitatively analyze the sample. If the sample is determined to contain asbestos at this stage, then it is quantitatively analyzed using the X-ray diffractometer. In actual practice, asbestos analysis (JIS A 1481) requires sensitivity sufficient to determine 0.1 % content by weight in 100 mg of acquired sample. To increase sensitivity so that such trace asbestos levels can be detected, formic acid is used to dissolve matrix components in the pulverized sample. Then the residue after formic acid treatment is recovered in a fluorocarbon polymer binder filter using a suction filtration system for use in quantitative analysis. In this case, the quantitative analysis is performed using an X-ray diffractometer, where the absorption of diffracted X-rays must be corrected to compensate for the asbestos itself and the undissolved matrix components. This correction process (base standard absorption correction method) involves first measuring the metal plate (base plate) for a blank filter, placing the filter with the formic acid-treated asbestos in the diffractometer, and measuring the asbestos together with the metal base plate to determine a correction factor from the diffraction intensity ratio of the metal plate. Then that correction factor is used to determine the corrected asbestos diffraction intensity. Note that this method was originally developed for measuring the free silicic acid content in mineral particulates during work environment measurements.

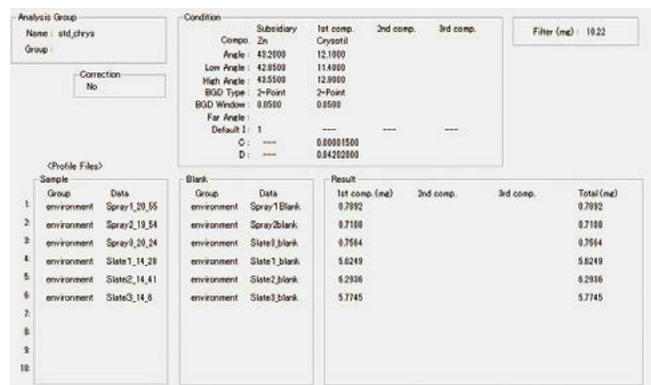
The XRD-6100 X-ray diffractometer environmental measurement package includes a user database for environmental samples, which is effective for increasing the accuracy of qualitative analysis, a filter holder and rotational sample stage for use in the base standard absorption correction method, and environmental quantitation software for performing quantitative calculations that correct for absorption. This environmental quantitation software incorporates Shimadzu's proprietary measurement expertise cultivated from many years in this field, which is especially valuable when quantitating particularly trace levels of asbestos. In addition, this XRD-6100 X-ray diffractometer environmental measurement package is compliant with methods specified in the Notification No. 0828001 by the Director of the Chemical Hazards Control Division, Industrial Safety and Health Department, Labour Standards Bureau, Ministry of Health, Labour and Welfare of Japan, such as for analyzing asbestos in natural minerals or measuring the free silicic acid content in mineral particulates during work environment measurements.



The major types of asbestos are serpentine asbestos (chrysotile) and amphibole asbestos (amosite and crocidolite). The characteristic peaks of these types can be quantitatively analyzed by X-ray diffractometry.



Qualitative analysis results for asbestos



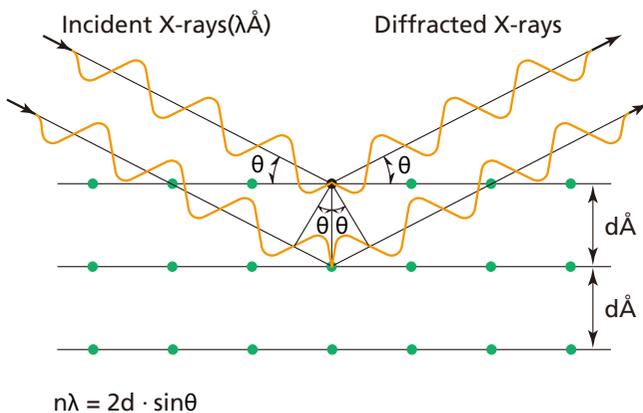
Screenshot of environmental quantitation software



# A general-purpose X-ray diffractometer to address your various analytical needs

## Principle of operation

The XRD-6100 analyzes crystalline states under normal atmospheric conditions. This method is non-destructive. X-rays focused on a sample fixed on the axis of the spectrometer (goniometer) are diffracted by the sample. The changes in the diffracted X-ray intensities are measured, recorded and plotted against the rotation angles of the sample. The result is referred to as the X-ray diffraction pattern of the sample. Computer analysis of the peak positions and intensities associated with this pattern enables qualitative analysis, lattice constant determination and/or stress determination. Qualitative analysis may be conducted on the basis of peak height or peak area. The peak angles and profiles may be used to determine particle diameters and degree of crystallization, and are useful in conducting precise X-ray structural analysis.



## Applications

Steels, non-ferrous metals, machinery, shipbuilding, welding, automobiles, ceramics, cement, glass, catalysts, electrical parts, electronic materials, magnetic materials, superconductive materials, fibers, paper, pulp, food products, chemicals, agricultural chemicals, dyes, pigments, paints, pharmaceuticals, dental materials, biological matter, petroleum, coal, power generation, natural gas, mining ore, soil, rocks, clay, minerals, construction, civil engineering, environment, and industrial waste.

## Construction

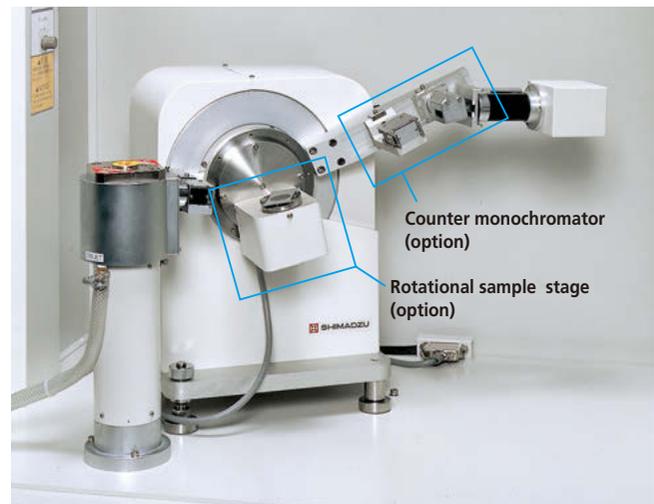
### Compact, X-ray-protected housing

The compact construction (W900xD700xH1600mm) minimizes installation space requirements.

The front door is mounted on guide rollers, enabling light and smooth opening of the door to facilitate easy installation/exchange of samples and attachments. A magnet latch assures the door closes; and to further ensure safety, a door interlock mechanism is automatically activated whenever X-rays are generated.

### High-precision, vertical goniometer

High-speed rate (1000°/min) and high-precision angle reproducibility ( $\pm 0.001^\circ$ ) provide fast measurement and highly reliable data. The vertical goniometer unit allows analysis of samples in various states, substantially widening the application range. The drive mechanism features an independent dual axis  $\theta$ - $2\theta$  linkage drive, and independent  $2\theta$  and  $\theta$  axis drives, freely selectable for efficient thin film and various other types of analysis.



Vertical Goniometer

### High voltage transformer for high output X-ray tube

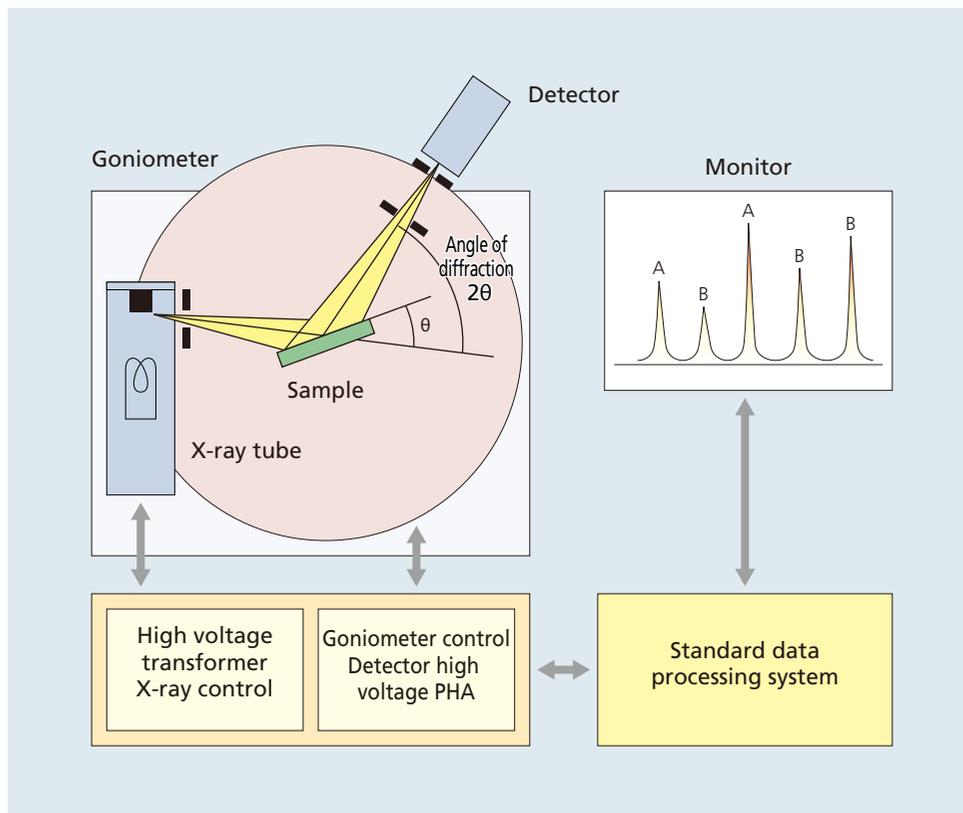
The high voltage transformer supports either the 2.2kW high output fine focus X-ray tube or 2.7kW high output broad focus X-ray tube.

### X-ray tubes

The XRD-6100 will accept various types of X-ray tubes, including the normal focus (NF) 2kW type and broad focus (BF) 2.7kW type, which are standard accessories, as well as the optional long fine focus (LFF) 2.2kW type. By attaching the optional counter monochromator, all types of samples, including Fe samples, can be analyzed using the standard Cu X-ray tube.

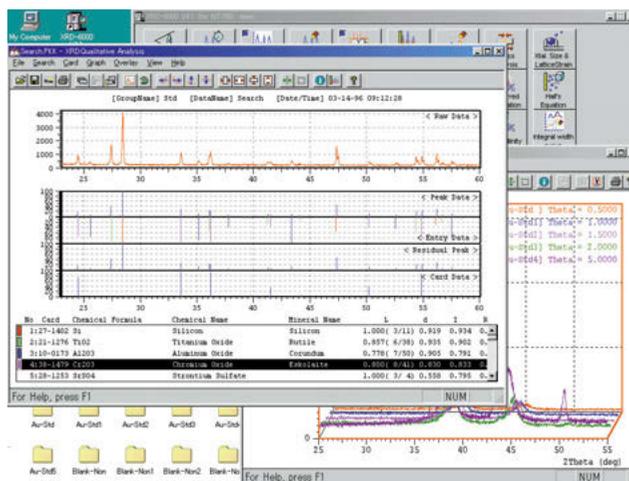
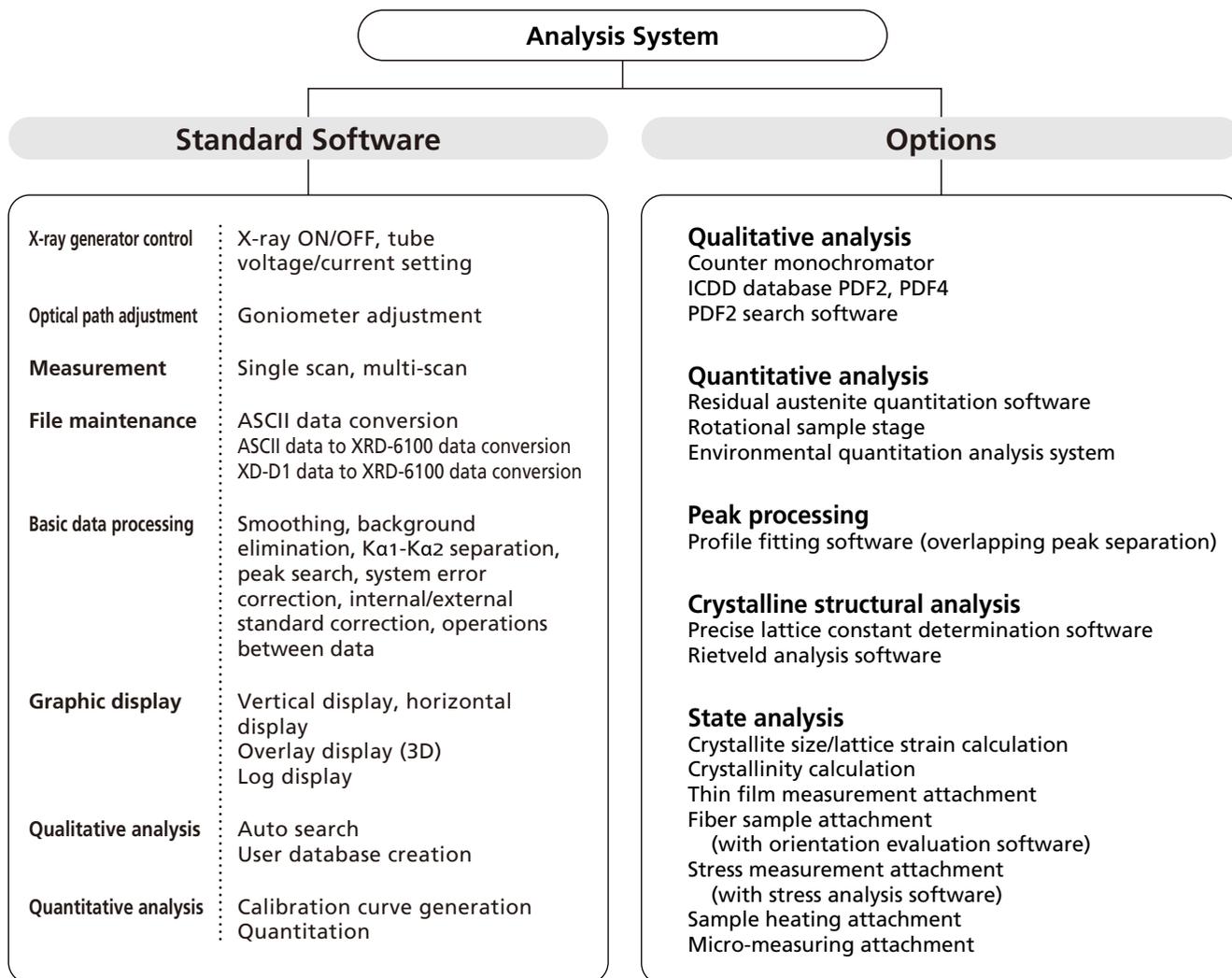
### Highly stable X-ray generator

Shimadzu's long experience in producing high-performance X-ray generators has enabled the production of a highly stable X-ray generator, with tube voltage and tube current both stable to within  $\pm 0.01\%$ . This stability is unaffected during fluctuation of source voltage or ambient temperature, ensuring high reliability of data even during prolonged periods of data acquisition.



XRD-6100 Relational Diagram

# Providing a Complete Analysis System



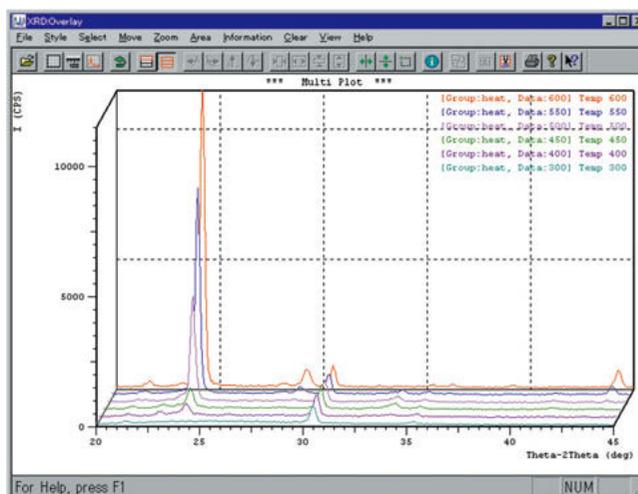
Auto search results and thin film sample overlay display



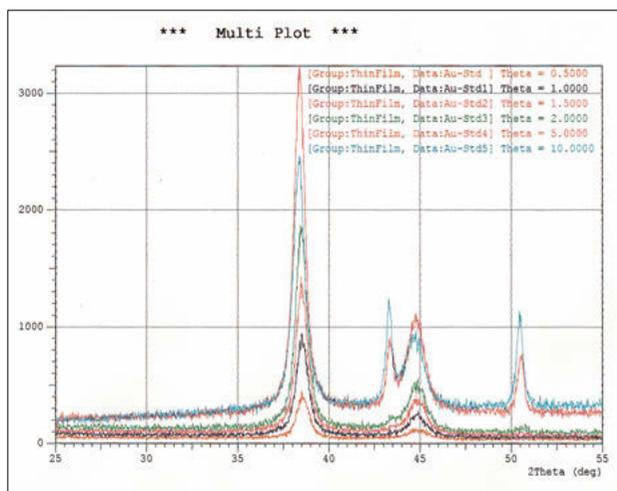
# Pleasant Data Processing Environment

## Graphic Display

Data can be freely zoomed with a click of the mouse, so profile comparison of thin film data or heating measurement data etc. is easily accomplished using combined 2-dimensional or 3-dimensional display. The software also features a variety of other useful graphic functions, such as intensity Log conversion display and hidden-line processing on the 3-dimensional display. Each type of data can be output to a color printer, so differences between samples can be recognized at a glance.



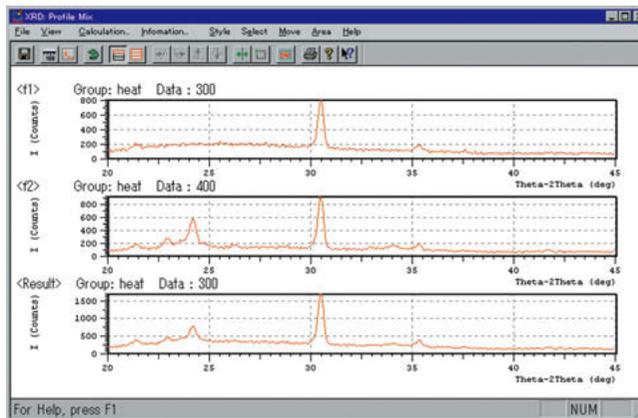
3-Dimensional Screen of Thin Film Sample



2-Dimensional Output of Thin Film Sample

## Adding/Subtraction Operations

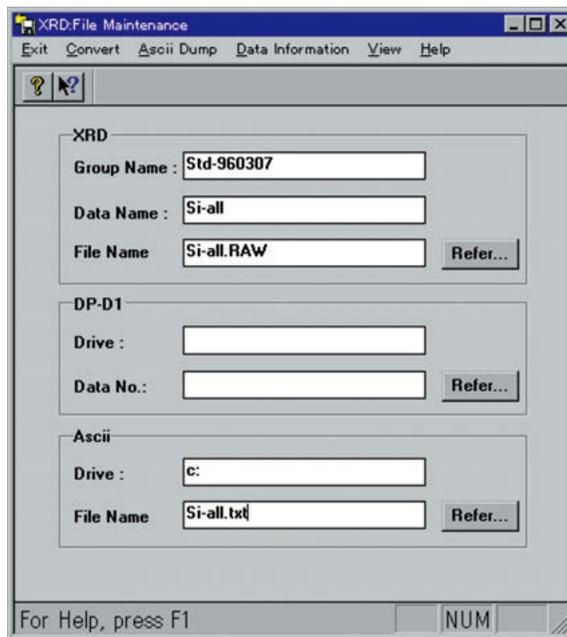
Data manipulation functions such as deletion of unnecessary peak profiles and addition of re-analyzed data to obtain a summed profile are some of the invaluable tools available for conducting efficient data analysis. Spectral calculations are conducted in the window displayed below.



Spectral Calculation Window

## File Maintenance ~Data Format Conversion~

Conversion between profile data and text data, conversion from Shimadzu X-ray Diffractometer XD-D1(previous model) acquisition data to XRD-6100 format data, and re-analysis are all possible. File format conversion is conducted using the window displayed at right.

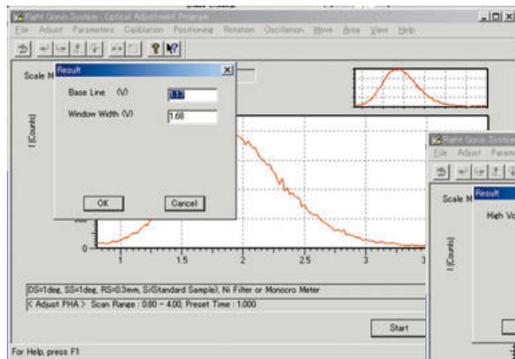


File Conversion Window

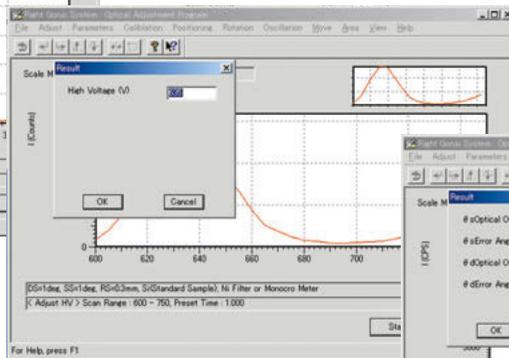
## Optical Adjustments

The XRD-6100 system makes fully automatic optical adjustments to the goniometer from the computer screen, even for optional attachments. In addition to completely automatically adjusting all settings, such as the zero angle for

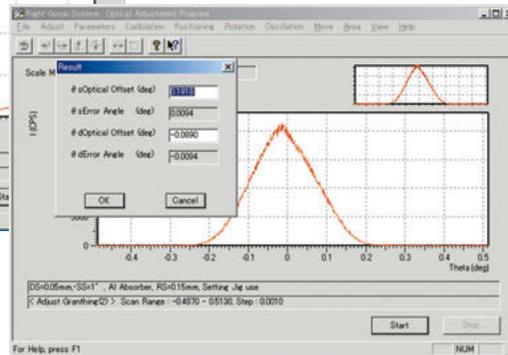
the  $\theta$  and  $2\theta$  axes, the x-ray detector high voltage settings, the PHA baseline and window width settings, it also automatically saves the settings information. This feature can be utilized for routine maintenance.



The PHA (Pulse Height Analysis) Adjustment Screen



HV (high voltage) adjustment screen



$\theta$ -Axis Adjustment Screen

# Enhanced Auto Search System

[ Auto Search, General Quantitation Software Provided as Standard ]

Identification work can be performed efficiently on screen.

## Detailed search parameters can be set.

To obtain correct results with automatic search/match, search parameters that conform to each sample must be set. The XRD-6100 enables the setting of detailed search parameters such as selection of files to be used in the search and three levels of element data input. Furthermore, the XRD-6100 comes with a standard function for element data, which takes up qualitative results (element analysis) from X-ray fluorescence spectrometers as files via LAN.

## Replete with second search function for authoritative identification of a small amount of components.

Identifying a small amount of components with a primary search is difficult; a second search is needed after the major components have been identified. The XRD-6100 is equipped with a second search function to provide an environment for easy identification of small amount of components.

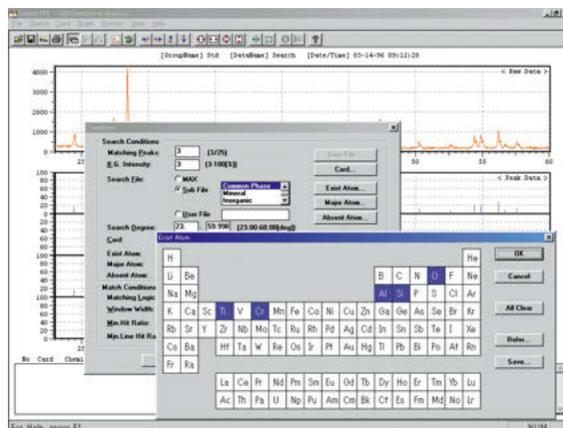
## Various search result data can be displayed.

Search results can be stack-displayed with each standard data display over raw data. Also, for easy comparison, standard substance names, chemical equations, ore names, Miller indices, and ICDD numbers can be displayed on each peak. Furthermore, an easy quantitative calculation function using a corundum ratio for candidate substances is included in the equipment.

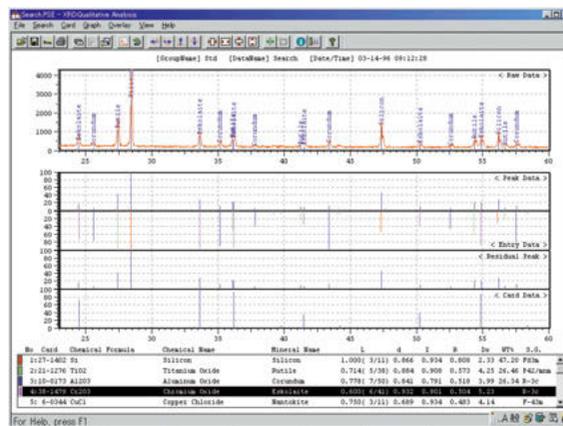
If your system has a PDF2 or PDF4 database, PDF2 or PDF4 detailed data for candidate substances can be displayed on a separate window.

## Dedicated user database can be created.

The user's own database file - separate from the sub-file supplied by ICDD (International Center for Diffraction Data) - can be created. Selected ICDD standard data and substance data not registered with ICDD can be input into this file. And data obtained through measurements by the XRD-6100 can be registered in the database file, which means that the user's basic samples can be registered, and comparisons made with those substances to provide an extra dimension to quality control.



Search Parameter Setting Screen



Search Result Screen

No.	JCPS #	Chemical Formula	Mineral Name	3-Strongest lines...d(1)	q.m.
1	10-0173	Al <sub>2</sub> O <sub>3</sub>	Corundum, syn	2.01(100) 1.10(23) 1.43(14)	*
2	33-0020	AlF <sub>3</sub>	Corundum, syn	2.01(100) 1.10(23) 1.43(14)	*
3	04-0707	Al	Aluminum, syn I	2.34(100) 2.02(47) 1.22(24)	*
4	04-0704	Au	Gold, syn	2.36(100) 2.04(52) 1.23(26)	*
5	04-0703	Ag	Silver-3ITC\CS	2.36(100) 2.04(48) 1.23(26)	*
6	38-1420	TiH	Sphorrite, syn	2.17(100) 2.43(72) 1.50(45)	*
7	06-0696	Fe	Iron, syn	2.03(100) 1.17(38) 1.43(28)	*
8	31-0619	(Fe,C)		2.00(100) 1.00(80) 1.00(80)	*
9	26-1078	C	Graphite-3ITB\	3.35(100) 2.00(11) 1.96(9)	*
10	11-0065	CuH	Calcite, #	2.07(100) 2.39(80) 1.46(80)	*
11	06-0694	Cr	Chromium, syn	2.04(100) 1.10(30) 0.91(20)	*
12	05-0727	Co		1.91(100) 1.25(80) 1.15(80)	*
13	04-0836	Cu	Copper, syn	2.05(100) 1.03(46) 1.20(48)	*
14	35-0821	Mg		2.45(100) 2.61(36) 2.70(25)	*
15	39-0794	HgD		3.05(100) 2.04(100) 1.17(100)	*

User Database Creation Screen

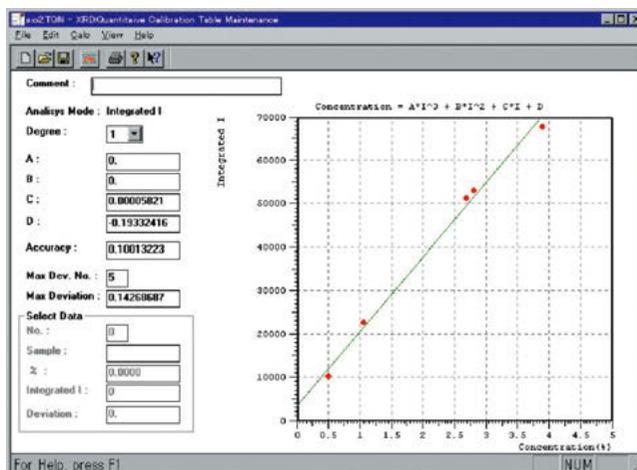
# Polished Quantitation Software

Satisfies your analysis objectives.

## Calibration Curves

Calibration curves can be generated for intensity, integrated intensity or intensity ratio.

Intensity and integrated intensity calculations are used for the internal standard and standard addition methods.

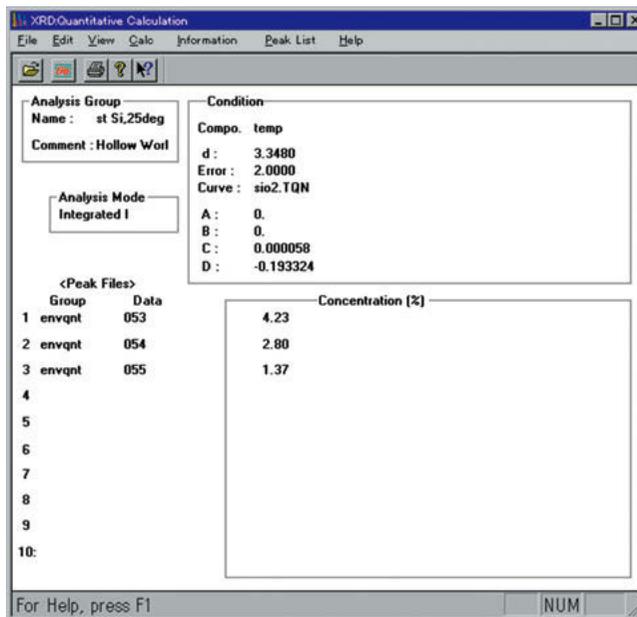


Calibration Curve Screen for Integrated Intensity

## Quantitative Analysis

The internal standard method and 2 intensity methods are available to satisfy most of the application needs.

Further, up to 5 peaks may be specified for quantitation and up to 10 sets of data may be calculated simultaneously.



Integrated Intensity Quantitation Results Screen

Note: Residual austenite quantitation and environmental quantitation software packages are optional.

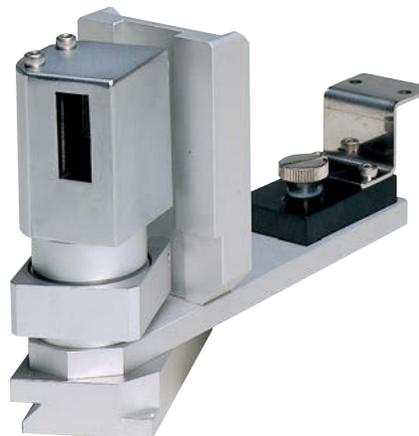
# Accessories

## Qualitative analysis

### Counter monochromator

Installed in the X-ray detector unit, the counter monochromator transforms X-rays which have passed through the entrance slit into monochromatic X-rays, allowing only the characteristic X-rays ( $K\alpha$  rays) to be detected. Exclusion of all other X-rays from the sample, including continuous rays and  $K\beta$  rays as well as fluorescent X-rays, ensures diffraction patterns with a high signal-to-noise ratio.

Part Description	Application	P/N
Counter monochromator CM-3121	Cu X-ray tube	215-22360-02
Counter monochromator CM-3131	Co X-ray tube	215-22360-03
Counter monochromator CM-3141	Fe X-ray tube	215-22360-04
Counter monochromator CM-3151	Cr X-ray tube	215-22360-05



### ICDD PDF2 / PDF4

This is the powder X-ray diffraction database provided by ICDD. PDF2 is provided on CD-ROM, and contains, in addition to substance name, chemical formula and d-I data, Miller indices, lattice constants, space groups and other crystallographic information. Using the special PDF2 Automatic Search Software (option), unknown substances can be easily identified via the registered crystallographic information.

ICDD PDF2	P/N for Educational Institutions	P/N for Other Uses
Single license <sup>Note1)</sup>	239-50002-12	239-50002-11

In addition to the functions of PDF2, database PDF4 features data searching software (DDVIEW+), the display of 2D, 3D structural charts, various lattice parameters, simulation wave form by the calculation, and the import of the measurement data. There are two databases of PDF4+ (for general) and PDF4/Organics (for organics).

ICDD PDF4+	P/N for Educational Institutions	P/N for Other Uses
Single license (New, 1 year license)	239-50015-02	239-50015-01
Single license (Renewal, 1 year license)	239-50015-04	239-50015-03
Single license (Renewal, 3 year license)	239-50015-06	239-50015-05
Single license (Renewal, 5 year license)	239-50015-08	239-50015-07

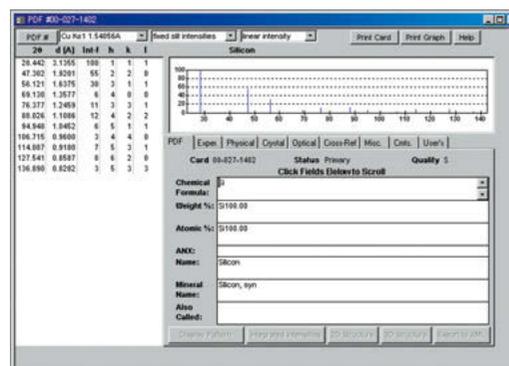
ICDD PDF4 / Organics	P/N for Educational Institutions	P/N for Other Uses
Single license (New, 1 year license)	239-50015-22	239-50015-21
Single license (Renewal, 1 year license)	239-50015-24	239-50015-23
Single license (Renewal, 3 year license)	239-50015-26	239-50015-25
Single license (Renewal, 5 year license)	239-50015-28	239-50015-27

### PDF2 Search Software

Searches can be performed from the card No., as well as based on multiple elements using "AND" or "OR" conditions, with analyte identification and crystalline structure obtained simultaneously.

PDF2 Search Software (DDVIEW)
Single license <sup>Note1)</sup>

Note 1: After 5 years, this license will need to be re-affirmed in order to make it perpetual.



## Qualitative analysis

### Rotational Sample Stage RS-1001

The RS-1001 performs in-plane rotation of the sample in combination with oscillation around the goniometer sample axis ( $\theta$ ) to minimize the scatter in diffraction pattern intensities attributable to the sample crystalline orientation, and thereby enhance the precision in most types of quantitative analysis.

#### Main specifications

- Rotation  $\beta$  axis (sample in-plane)
- Rotation speed 1 to 60rpm
- Minimum step width 0.1 degree
- Operation modes Constant speed rotation, oscillation sample in-plane rotation scan (continuous, step)
- Measuring angle range  $2\theta$  5° to 163°



Part Description	P/N
Rotational sample stage (without option driver)	215-21766-01

Note: Please arrange the option driver at the same time.  
Please refer to the special accessories on page 24.

### Environmental Measurement Stage RS-2001

A complete environmental analysis system, this comprises a special environmental quantitative analysis stage, filter holder and quantitation software. A special filter holder is provided which allows measurement using an asbestos-imbedded filter as is. The main specifications of the environmental stage are the same as those of the general-purpose rotational sample stage. The calibration curve correction is based on Zn; however, when the diffraction line of the sample overlaps with that of Zn, an Al sample holder (optional) is also available. The sample stage option driver can also be used with the rotational sample stage.



#### Main specifications

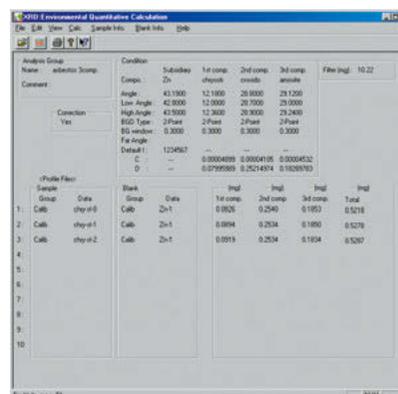
- Measuring angle range  $2\theta$  5° to 163°

Part Description	P/N
Environmental Analysis Stage (with S/W)	215-21767-03
Al filter holder ( $\phi$ 25)	215-23765-92
Aluminum sample holder (5PC)	215-22507-06

Note: Please arrange the option driver at the same time.  
Please refer to the special accessories on page 24.

### Environmental Quantitation Software (P/N 215-00421-92)

Environment samples, as suspended dust particles, collected in a very small quantity on a filter present an analytical challenge. The XRD-6100 reliably addresses this challenge. The software eliminates the effect of X-ray absorption by the filter, providing a calibration curve with good linearity and high accuracy. The software associated with the use of a special sample holder allows the application of a very efficient filter absorption correction.



# Accessories

## Automatic Analysis

### Auto 5 Position Sample Changer ASC-1001

This stage is used in order to automatically measure a maximum of 5 samples.

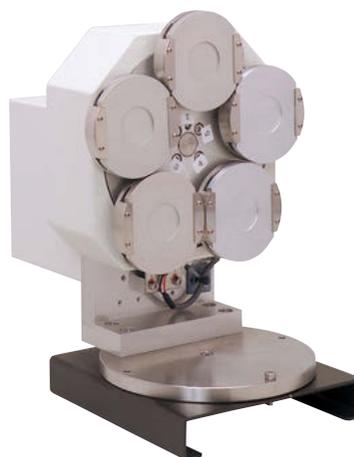
The ASC-1001 performs in-plane rotation of the sample in combination with oscillation around the goniometer sample axis ( $\theta$ ) to minimize the scatter in diffraction pattern intensities attributable to the sample crystalline orientation. It is also possible to use the filter holder (option) with the Environmental Measurement Stage RS-2001.

#### Main specifications

- Sample position 5
- Sample size Powder: 25mm  $\phi$   
Filter: 25mm  $\phi$  (option)
- Rotation speed 1 to 60rpm
- Measuring angle range  $2\theta$  5° to 163°

Part Description	P/N
Auto 5 position sample changer (with an option driver unit)	215-23175-01
Zn filter holder (25mm $\phi$ ) 5pc/set	215-23760-91
Al filter holder (25mm $\phi$ ) 5pc/set	215-23760-92

Note: Please arrange the option driver at the same time.  
Please refer to the special accessories on page 26.



### Sample Plates for RS-2001 and ASC-1001

Part Description	P/N
Aluminum sample holder (5pc)	215-22507-06
Glass sample holder (5pc)	215-22507-07
Glass Micro sample holder (5pc)	215-22507-08
Non-reflective sample holder (2pc)	215-22507-09

### Thin Film Analysis using Attachment THA-1101

This is a specialized thin film analysis that includes the thin film sample stage, monochromator and suction pump.

Employing the fixed incidence angle, parallel X-ray diffractometry method, penetration of incident X-rays into the substrate sample is limited as much as possible, providing low background, thin film X-ray diffraction patterns.

Specimens are easily set in place using the suction pump.

The sample stage option driver can also be used with the rotational sample stage.

#### Main specifications

- Rotation  $\beta$  axis (sample in-plane)
- Rotation speed 1 to 60rpm
- Minimum incidence angle 0.1degree
- Sample suction pump AC100V, 10W (1 pump)
- Operation modes Constant speed rotation, oscillation, sample in-plane rotation scan, (continuous, step)

Part Description	P/N
Thin film analysis attachment (without option driver)	215-21765-01

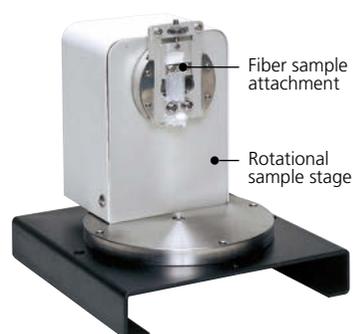
Note: Please arrange the option driver at the same time.  
Please refer to the special accessories on page 26.



## Fiber Sample Attachment

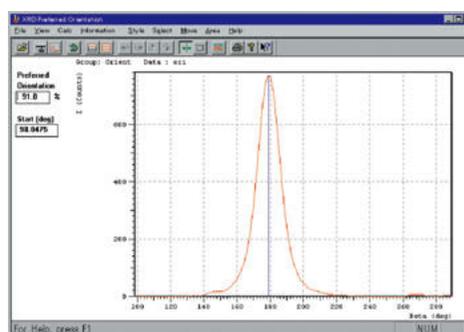
Used in combination with the Rotational Sample Stage (RS-1001), this system measures the degree of orientation for fibers. The acquired data is then processed using the provided fiber sample attachment software to calculate the degree of orientation.

Part Description	P/N
Fiber sample attachment (with S/W)	215-22624



## Fiber orientation software (P/N 215-00428-92)

This software evaluates the degree of orientation for fiber samples, using the data of peak width at half height acquired from orientation measurement (sample in-plane  $\beta$  axis measurement).



Degree of Orientation Evaluation Screen

## Automatic Variable Slit System AVS-1101

Environment samples, as suspended dust particles, collected in a very small quantity on a filter present an analytical challenge. The XRD-6100 reliably addresses this challenge. The software eliminates the effect of X-ray absorption by the filter, providing a calibration curve with good linearity and high accuracy. The software associated with the use of a special sample holder allows the application of a very efficient filter absorption correction.

This mechanism automatically sets the DS, SS and RS slit widths according to the measurement mode selected on the screen.

- **Mode:**  
The emission slit is adjusted so that all sample surfaces are irradiated with the X-ray of the same width. The detector slits (SS and RS) are also adjusted in accordance with the irradiation width.
- **Fixed Irradiation Width Mode:**  
The DS, SS and RS slit widths are fixed at the set values.

The data obtained using this software can be converted to the conventional fixed-slit-width data by performing irradiation width compensation (patent pending).

Part Description	P/N
AVS-1101 Automatic Variable Slit System	215-23950



Degree of Orientation Evaluation Screen

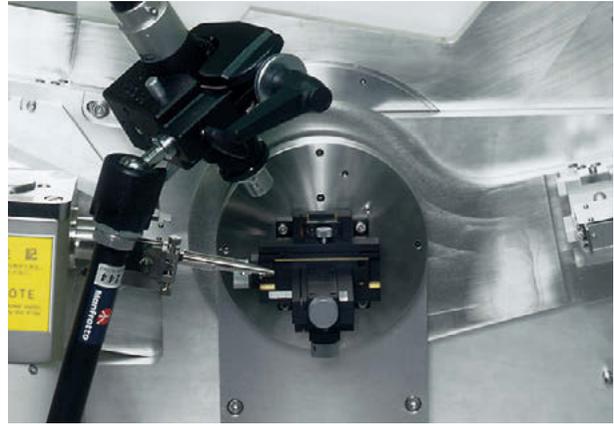
# Accessories

## Micro Area Measurement Attachment MDA-1101/1201

The Micro Area Measurement Attachment uses a pinhole slit for emission, allowing the measurement of micro regions. Measured surfaces are observed via a CCD camera, so observation images can be loaded onto a computer, saved and edited. The product line includes two models: the MDA-1101 that uses an optical microscope and the MDA-1201 that uses a zoom (8 - 80 mm) camera lens.

### Key Specifications

- Pinhole Emitter Slit                    0.1, 0.2, 0.3, 0.5, 1, or 2 mm diameters
- XYZ Movement                         ±7.5 mm
- Sample Surface Observation Method    CCD camera image viewed on computer screen



Part Description	P/N
Micro Area Measurement Attachment (MDA-1101)	215-23180-93
Micro Area Measurement Attachment (MDA-1201)	215-23180-94

## Stress Analysis Attachment SA-1101

This specialized stress analysis system using the side-inclination method includes the stress analysis sample stand, X-ray tube and stress, analysis software.

X-ray stress analysis is widely used to measure the level of stress in substances.

In the X-ray diffractometry of stress extremely small changes in the lattice space are measured from the X-ray diffraction pattern profile. The use of the special stress analysis stand associated with the side-inclination method allows the precise measurement of the residual stress. This technique is free of absorption error.

The software includes the following functions: as measurement, width at half height, peak position calculation and stress calculation. Depending on the type of sample and reflective plane, either the Cr X-ray tube or Co tube is necessary. The sample stand option driver can also be used with the rotational sample stage.



### Main specifications

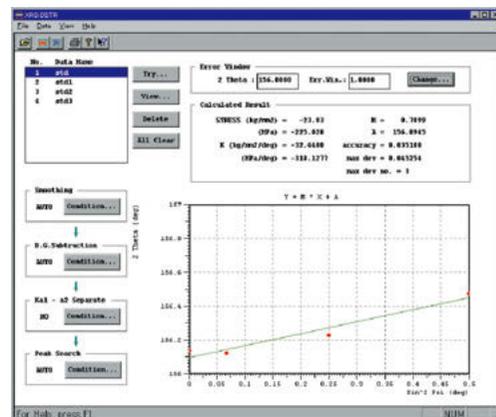
- Inclined axis                             $\alpha$  axis
- Inclined angle range                 0 to 50 degrees
- Operation modes                        Oscillating, fixed

Part Description	P/N
Stress analysis attachment (with Cr tube, S/W)	215-21769-01
Stress analysis attachment (with Co tube, S/W)	215-21769-03

Note: Please arrange the option driver at the same time.  
Please refer to the special accessories on page 26.

## Stress Analysis Software (P/N 215-00429-92)

This software can analyze data obtained using either a parallel-beam (fixed  $\psi$  or fixed  $\psi$  0) or orthogonal-beam method.



Residual Stress Analysis Result Screen

## PCL-1001 Polycapillary Unit

The polycapillary unit is a new optical X-ray element that splits a single X-ray beam emitted from a point's light source into multiple X-ray beams using three-dimensionally arranged capillary optics. This creates a powerful parallel beam output that covers a large area.

1) Compared to conventional methods, this unit uses the X-ray more effectively and increases the intensity of the diffracted X-ray, allowing more sensitive analysis.

2) With conventional methods, variations in sample surface height are directly translated into variations in X-ray diffraction angles. This polycapillary unit uses parallel beams, so it is not affected by variations in sample surfaces.

Part Description	P/N
PCL-1001 Polycapillary Unit	215-23980
CM-4121 Counter Monochromator Assembly (for parallel beams)	215-22360-06
X-Ray Tube (Long fine focus, with Cu target)	210-24100-11

Note: If an LFF type X-ray tube is used in the XRD-6100 system, the X-ray tube listed above is not required.



## Sample Heating Attachment HA-1001

This system, consisting of a special sample heating furnace and temperature controller, is used to heat the sample during X-ray diffractometry to study the influence of heat on the crystalline structure. The atmosphere in the furnace, consisting of air, an inert gas or a vacuum, may be heated to 1500°C during measurement. The measurement results are output in multiple data formats to enable comparison of X-ray diffraction patterns obtained at various temperatures.

### Main Specifications

- Thermocouple Pt-Pt/Rh
- Measurement temperature 1500°C max. in vacuum, air  
1200°C max. using inert gas (N<sub>2</sub>)
- Control functions PID value setting, fixed temperature control  
(temperature increase, decrease, hold, stop)
- Power supply Single phase 200/220V±10% 10A

Part Description	P/N for Other Uses
Sample heating attachment (for XRD-6100 with temperature controller)	215-23000



HA-1001

# Accessories

## Heating or Cooling Attachment TTK-450

This system, consisting of a special sample heating furnace and temperature controller, is used to heat the sample during X-ray diffractometry to study the influence of heat on the crystal structure. The atmosphere in the furnace, consisting of air, an inert gas or vacuum, may be heated to 450°C during measurement at TTK-450.

With a vacuum kit and cooling kit, the atmosphere may be cooled to -180°C. at TTK-450

The measurement results are output in multiple data formats to enable comparison of X-ray diffraction patterns obtained at various temperatures.

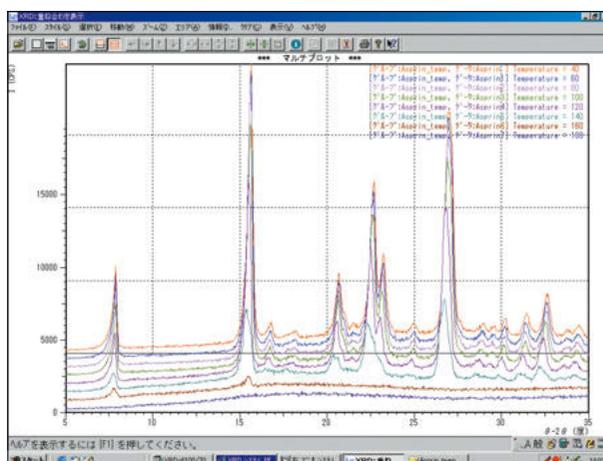
### Main Specifications

	TTK-450
Thermocouple	PT100 resistor
Power supply Temperature	RT to 300°C (in the air, or an inert gas) RT to 450°C (in vacuum) -180 to 450°C (With cooling kit in vacuum)
Control functions	PID value setting fixed temperature control (increase, decrease, hold, stop)
Power supply	single phase 200/220V±10% 5A

Part name	P/N
Heating attachment TTK-450	215-24030-93
Vacuum kit for TTK-450 for XRD-6100	215-24034-91
Cooling kit for TTK-450 for XRD-6100	215-24033-91



TTK-450



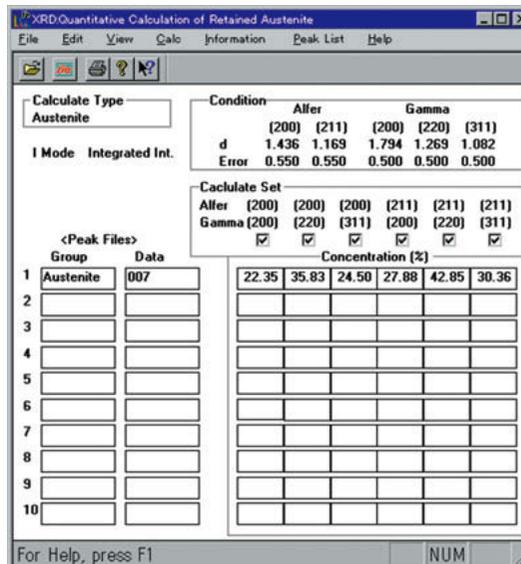
Sample Heating Measurement Result Screen

## Qualitative analysis

### Residual Austenite Quantitation (P/N 215-00430-92)

A common method to quantify residual austenite is to apply the method for samples consisting of 2 components such as tempered copper  $\alpha$ -iron and  $\gamma$ -iron. Special software allows the determination without the need of standard sample.

The software directly uses the intensity ratio of the measured X-ray peaks of the  $\alpha$ -iron and  $\gamma$ -iron components to theoretically perform the calculation. The five-peak average method is used to make the determination, so scattering due to the matrix effect is reduced, enhancing the reliability of the results. Using the rotational sample stage (P/N 215-21766) for measurement further helps to overcome data scattering.



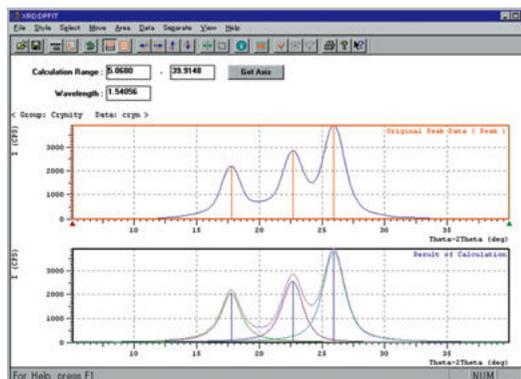
Quantitation Results Screen

## Peak Processing

### Overlapping Peak Separation Software (P/N 215-00423-92)

Using the Gauss and Lorentz models, overlapping peaks are separated one by one, with information including position, intensity, width at half height and integrated intensity calculated for each diffraction peak.

These are then utilized to conduct quantitative analysis and crystalline structure analysis.

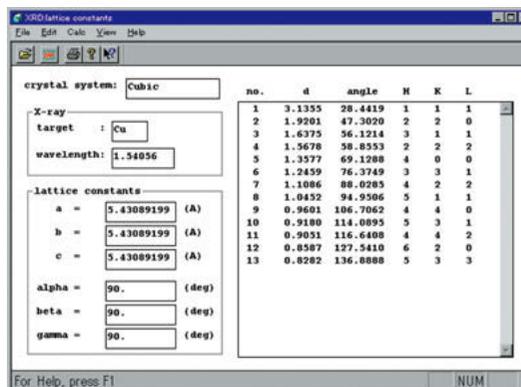


Peak Separation Screen

## Crystalline Structural Analysis

### Precise Lattice Constant Determination Software (P/N 215-00424-92)

In X-ray diffractometry, higher accuracy is often required to determine the lattice constant, which is a fundamental parameter for determining a substance's crystalline structure. This is most often used for quantitating solid solution content. This software corrects the raw diffraction angle data calculated via basic data processing to determine enhanced precision lattice constants for up to 7 crystals concurrently, employing the least squares method to further minimize error in diffraction angles. In addition, the Miller index is applied to each peak.



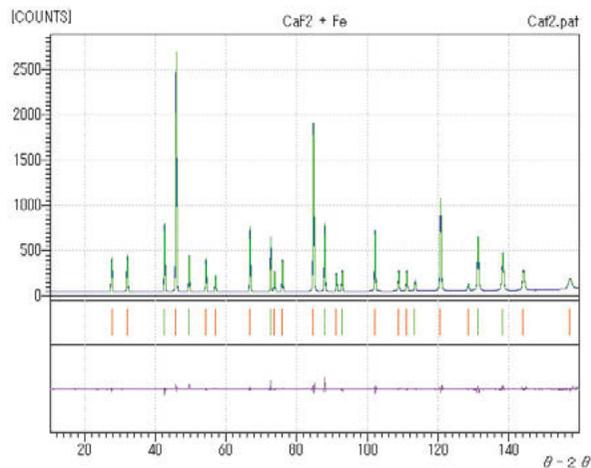
Precise Lattice Constant Determination Calculation Result Screen

# Optional Software

## Crystalline Structure Analysis

### Rietveld Analysis Software (P/N 215-00434-92)

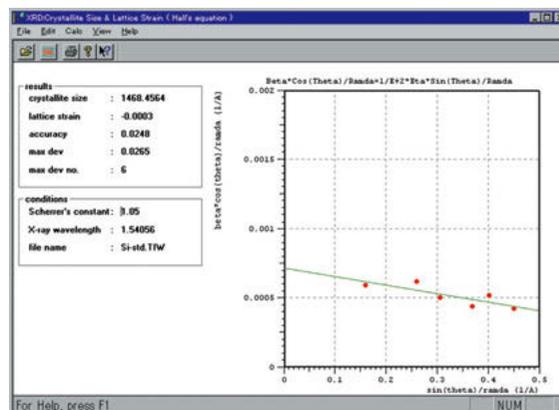
The Rietveld method analyzes the crystalline structure by directly refining structural parameters and lattice constants over the entire powder X-ray or neutron diffraction pattern. It compares the diffraction pattern calculated from a presumed structural model with the actual measured pattern, and refines each parameter using the nonlinear least square method developed by the National Institute for Materials Science (formerly the Institute for Research in Inorganic Materials). This Rietveld Analysis Software utilizes the RIETAN program created by Mr. Fujio Izumi at the National Institute for Materials Science.



## State Analysis

### Crystallite Size & Lattice Strain Software (P/N 215-00426-92)

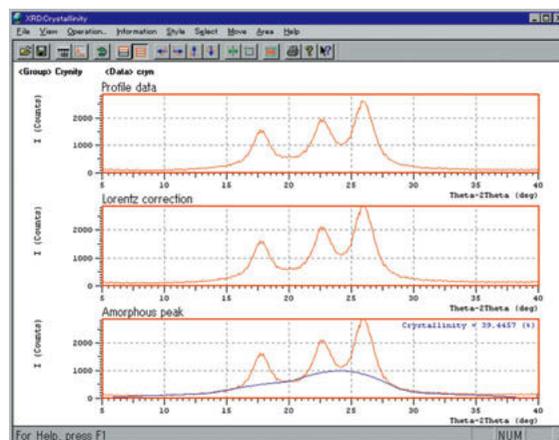
Samples normally consist of crystallites ranging in size from several  $\mu\text{m}$  to tens of  $\mu\text{m}$ . However, in the case of catalyst crystallites, which may measure several hundred  $\text{\AA}$ , X-ray diffraction is insufficient, resulting in diffraction peak spreading. This software quantitatively determines that spread, and applies the Scherrer's equation to calculate the crystallite size. When there is involvement of lattice strain, the diffraction spread is determined for a number of diffraction peaks, and from the resultant line slope and intercepts, the size of each of the crystallites and the lattice strain are calculated. (Hall's Method)



Hall's Equation Calculation Result Screen

### Crystallinity Calculation Software (P/N 215-00427-92)

The degree of crystallization of a mixture of crystalline and amorphous substances, such as found in high polymer samples, is an important parameter of substance characterization. This software automatically or manually separates the measured diffraction patterns into those of crystalline components and those of amorphous components. Then, it calculates the integrated intensity of the two types of substances, called degree of crystallization using the peak area ratio of the two classes of components.



Crystallinity Calculation Result Screen

# Other Accessories

## Sample Holders

The following sample holders, including the aluminum sample holder, which is supplied as standard with the diffractometer, are available for different applications.



Part Description	Sample area	Application	Remarks	P/N
Aluminum Sample Holder	ø25 (dia.) x 1mm (d)	General purpose	Made of aluminum, 5pc	215-22507-01
Glass Sample Holder	ø25 (dia.) x 1mm (d)	Lattice constant	Made of glass, 5pc	215-22507-02
Glass Micro Sample Holder	ø15 (dia.) x 0.5mm (d)	Micro samples	Made of glass, 5pc	215-22507-03
Non-reflective Sample Holder		Ultramicro samples	Made of silicon, 2pc	215-22507-05

## X-ray Tubes and X-ray Filters

Focus Type	Type NF	Type BF	Type LFF
Focus Size	1 x 10mm	2 x 12mm	0.4 x 12mm
Tube voltage, current	60kV, 50mA	60kV, 60mA	60kV, 55mA
Target	X-ray Tube Maximum Load & P/N		
Cu	2.0kW (239-24014-01)* <sup>1</sup>	2.7kW (210-24016-21)	2.2kW (210-24100-11)
Co	1.8kW (062-40003-04)	2.7kW (210-24016-24)	1.8kW (210-24100-14)
Fe	1.5kW (062-40003-05)	2.2kW (210-24016-25)	1.0kW (210-24100-15)
Cr	2.0kW (062-40003-06)	2.7kW (210-24016-26)	1.9kW (210-24100-16)

X-Ray Filter	
Part Description	P/N
Ni filter (for Cu)	(215-22500-02)
Fe filter (for Co)	(215-22500-03)
V filter (for Cr)	(215-22500-05)

\*1 When using it as a point focus, combine a X-ray tube and a point focus head of following P/N.

1) X-ray tube (Cu target, 2.0kW, NF) P/N 210-24016-11

2) point focus head P/N 239-16047

\*When using the polycapillary system, use LFF type.

## Cooling Water Circulator

RKE1500B-V-G2-SP  
(for 3kW X-ray tube)

With its built-in cooler, the Cooling Water Circulator cools the X-ray tube and X-ray generator by circulating cooled, pure or clean water. The unit is recommended when no tap water is available or the available water is of poor quality.

### Main specifications

- Power supply Three phase 200V ±10% 10A (RKE1500B-V-G2-SP)
- Ambient temperature 5 to 40°C
- Cooling capacity 5.3kw/h (50/60Hz) (RKE1500B-V-G2-SP)



RKE1500B-V-G2-SP

Part Description	P/N
RKE1500B-V-G2-SP	239-15049-02

# Accessories

## Special Accessories

◎: Absolutely required   ○: Required   ●: Required depending on objective

Analysis Objective	Part Description	Part Number	Iron and steel related	Non-ferrous metals, precious metals	Machining, automotive stamping, welding	Brickmaking, ceramics	Cement and glass	Electrical, electronic materials	Foodstuffs, textiles, paper, pulp	Chemicals, catalysts, dyes, paints	Medical, dental materials, biological operations	Natural resources, energy	Construction, engineering	Environment, industrial waste	Environment (Asbestos)
1 BG reduction, especially iron samples	Counter Monochromator CM-3121	P/N 215-22360-02	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
2 Qualitative analysis PDF2 Search	ICDD PDF2 file (CD-ROM)	P/N 239-50002-11,12	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
	PDF2 Search S/W (DDVIEW)	P/N 239-50002-21,22													
3 Qualitative analysis PDF4 Search	ICDD PDF4 + (CD-ROM)	P/N 239-50015-01,02	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
4 General purpose quantitative analysis	*Rotational Sample Stage RS-1001 <sup>Note1</sup>	P/N 215-21766-01	○	○	○	○	○	○	○	○	○	○	○	○	○
	Auto 5 position sample changer ASC-1001 <sup>Note1,2</sup>	P/N 215-23175-02													
5 Residual austenite quantitation	*Residual austenite quantitation S/W	P/N 215-00430-92	○		○										
	*Rotational Sample Stage RS-1001 <sup>Note1</sup>	P/N 215-21766-01	○		○										
6 Environmental quantitative analysis system	*Environmental Quantitative Analysis Stage RS-2001 <sup>Note1</sup> (Filter holders Zn, with S/W)	P/N 215-21767-01												◎	◎
	Filter holder Al (ø 25mm)	P/N 215-23765-92												◎	◎
7 Multiple peak separation	Profile fitting S/W	P/N 215-00423-92	●	●	●	●	●	●	●	●	●	●	●	●	●
8 Precise lattice constant determination	Precise lattice constant determination S/W	P/N 215-00424-92	●	●	●	●	●	●		●					
9 Crystal structure analysis	Rietveld analysis, RIETAN software	P/N 215-00433-92	●	●	●	●	●	●		●					
10 Crystallite size / lattice stress	Crystallite Size / Lattice Stress S/W	P/N 215-00426-92							○	○	○				
11 Degree of crystallization	Degree of Crystallization S/W	P/N 215-00427-92	●						○	○	○				
12 Heating analysis	Sample Heating Attachment HA-1001	P/N 215-23000-01	●	●	●	●	●	●		●	●				
13 Thin film analysis	*Thin Film Analysis Attachment THA-1101 <sup>Note1</sup> (stage, monochromator, suction pump)	P/N 215-21765-01	●	●	●	●	●	●							
14 Fiber degree of orientation analysis	Rotational Sample Stage RS-1001 <sup>1)</sup>	P/N 215-21766-01	●	●	●	●	●	●	○	●					
	Fiber Sample Attachment (with S/W)	P/N 215-22624	●					●	○	●					
15 Residual stress analysis	*Stress Analysis Attachment SA-1101 <sup>Note1</sup> (with Cr X-ray tube, S/W)	P/N 215-21769-01	○	○	○	●	●	●							
16 Micro Measurement with microscope	Micro-Measuring Attachment MDA-1101	P/N 215-23180-93	●	●	●	●		●		●					
17 Micro Measurement with CCD camera	Micro-Measuring Attachment MDA-1201	P/N 215-23180-94	●	●	●	●		●		●					
18 Auto Mapping (stress, quantitative analysis)	R-θstage for large sample		●	●	●	●	●	●	●	●	●	●	●	●	●
19 Strong parallel beam X-ray source	poly-capillary unit PCL-1002	P/N 215-24375-91	●	●	●	●	●	●	●	●	●	●	●	●	●

1) Can be used together with general purpose rotational sample stage

Note1: Please arrange optional additional ASSY(P/N215-23705) with optional driver ASSY(P/N215-21764) at the same time when you arrange the accessories of the asterisk. Moreover, even when two or more accessories are arranged, the option driver ASSY and optional additional ASSY can use it combinedly with one unit.

Note2: When I arrange an auto 5 position sample changer, please arrange two optional additional ASSY.

# Specifications

## XRD-6100

Item	XRD-6100			
	2kW Type	3kW Type	High-resolution type	
X-ray tube	Type	Cu, NF type	Cu, BF type	
	Focus	1.0 x 10mm	2.0 x 12mm	
	Max. output	2kW	2.7kW	
X-ray generator	Max. output	3kW		
	Output stability	±0.01% (for 10% power fluctuations)		
	Max. tube voltage	60kV		
	Max. tube current	80mA		
	Voltage step width	1kV		
	Current step width	1mA		
	Overload limit setting	Setting changeable with tube type		
	X-ray tube protection	Against undervoltage, overload, overvoltage, overcurrent and/or failure of water supply		
	Safety mechanisms	Door interlock mechanism (X-ray can be generated only after the door is closed) Emergency stop		
	Goniometer	Type	Vertical type	
Scanning radius		185mm		
Min. step angle		0.002° (2θ) 0.001° (θ)		
Angle reproducibility		±0.001° (2θ)		
Scanning angle range		-6° to 163° (2θ), -180° to 180°(θ)		
Scanning system		θ/2θ linkage mode, θ, 2θ independent mode		
Operation mode		Continuous scan measurement, step scan measurement, calibration, positioning, θ axis oscillation (when using 2θ continuous scan or step scan)		
Max. speed		1000°/minute (2θ)		
Scanning speed		0.1° to 50°/min (2θ), 0.05° to 25°/min (θ)		
Divergence slit (DS)		0.5°, 1°, 2°, 0.05mm		
Scattering (SS)		0.5°, 1°, 2°		
Receiving slit (RS)		0.15mm, 0.3mm		
Casing		Dimensions	W900 x D700 x H1600	
		Leakage X-rays	Less than 2.5μSv/h (at maximum output)	

## X-ray Tubes and X-ray Filters

Focus Type	Type NF	Type BF	Type LFF
Focus Size	1 x 10mm	2 x 12mm	0.4 x 12mm
Tube voltage, current	60kV, 50mA	60kV, 60mA	60kV, 55mA
Target	X-ray Tube Maximum Load & P/N		
Cu	2.0kW (239-24014-01)*2	2.7kW (210-24016-21)	2.2kW (210-24100-11)
Co	1.8kW (062-40003-04)	2.7kW (210-24016-24)	1.8kW (210-24100-14)
Fe	1.5kW (062-40003-05)	2.2kW (210-24016-25)	1.0kW (210-24100-15)
Cr	2.0kW (062-40003-06)	2.7kW (210-24016-26)	1.9kW (210-24100-16)

X-Ray Filter	
Part Description	P/N
Ni filter (for Cu)	(215-22500-02)
Fe filter (for Co)	(215-22500-03)
V filter (for Cr)	(215-22500-05)

\*2 When using it as a point focus, combine a X-ray tube and a point focus head of following P/N.

- 1) X-ray tube (Cu target, 2.0kW, NF) P/N 210-24016-11
- 2) Point focus head P/N 239-16047

\*When using the polycapillary system, use LFF type.

## Detectors

### OneSight Wide-Range High-Speed Detector

(FD-1001 1D High-Speed Detector P/N 215-24320-91)

Scan range	0-159° : Radius of Goniometer 275 mm 0-150° : Radius of Goniometer 200 mm 0-148° : Radius of Goniometer 185 mm
Operation mode	Step-scan mode, One-shot mode
Sensor	Reverse biased pn-junction array
Detection principle	Single photon counting
Weight	280 g
Active area	64 x 8 mm
Number of channels	1280
Width of one channel	50 μm

### Scintillation Detector

SC-1003 P/N 215-24385-92

Scintillator	Na I
Scaler	Preset time: 0.1 to 1000s; digits: 7
HV/PHA	500 to 1200 V high-voltage power supply, baseline and window auto-controlled

## Data Processing Unit

Computer	IBM PC/AT compatible
OS	Windows 7
Controlled elements	Goniometer, X-ray generation, tube voltage, tube current, detector high voltage, PHA, scaler
Basic data processing	Smoothing, BG elimination, Kα1-Kα2 separation, peak searching, peak width at half height, integrated intensity, systematic error correction, internal/external standard correction, operations between data, graphic display
Qualitative analysis	Database (library) creation, automatic library search (ICDD PDF2/PDF4 options) calibration curve generation, quantitation calculation
Quantitative analysis	Calibration curve generation, quantitative analysis

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\*Additionally noted company names and product names are the trademarks or registered trademarks of the respective companies.

\*The notations™ and ® are not used in this document.

# Installation Requirements

## Installation Site

This instrument uses X-rays for measurement and analysis. Accordingly, before installing the instrument, be sure to consult local regulations regarding measures associated with X-ray generation, and comply with all necessary regulatory procedures.

### Power requirements

For main unit	Single phase 200/220V $\pm 10\%$ 2kW type: 30A 3kW type: 50A
Data processing unit	Single phase 100V 10% $\pm 10A$
Ground	Independent, at least 100 $\Omega$ resistance

### Installation site environment

The following ambient temperature and humidity are required.

Temperature	23°C $\pm 5^\circ\text{C}$
Humidity	60% $\pm 5\%$

Heat generated from the instrument is approximately 1kW/h. When the cooling water circulator is installed in the same room, this is increased by 3.2kW/h and 5.3kW/h for the 2kW and 3kW types, respectively.

### Cooling water supplied to instrument

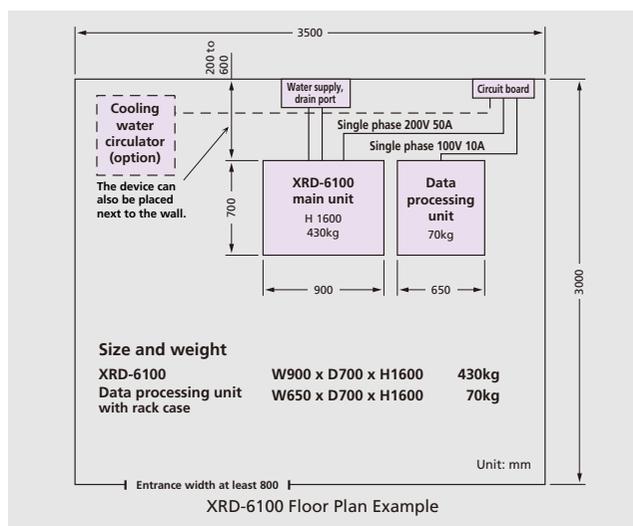
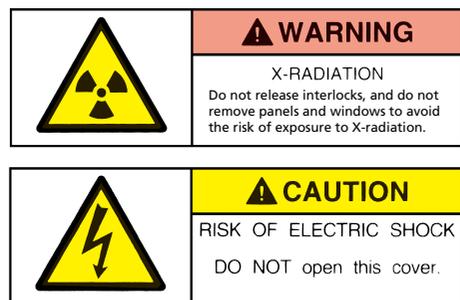
When cooling water supplied to the instrument becomes dirty due to piping corrosion, etc., this causes clogging of the X-ray tube filters. Cooling water should be supplied using the following conditions.

Temperature	23°C $\pm 5^\circ\text{C}$
Humidity	60% $\pm 5\%$

Avoid any sudden changes in temperature, which might cause condensation to form on the surfaces of internal parts. Heat generated from the instrument is approximately 860cal/h. When the cooling water circulator is installed in the same room, this is increased by 3.2kW/h for the 2kW X-ray tube and 5.3kW/h for the 3kW X-ray tube.

Flow rate	At least 4.0L/min
Water pressure	3 to 5kgf/cm <sup>2</sup>
Water quality	pH6 to 8, hardness less than 80ppm
Particulates	Less than 0.1mm
Supply water port diameter	12.7mm $\phi$
Drain water port	Natural drainage

If the flow rate is lower than 4.0L/min, the safety circuit for protection of the X-ray tube is active, disabling the X-ray generation circuit. When minimum conditions of the flow rate can not be fulfilled, use the cooling water circulator, available as an option.



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